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Magnetic force microscopy study of magnetization reversal in sputtered FeSiAl(N) films

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The magnetization reversal in a series of rf-sputtered FeSiAl(N) films has been studied using magnetic force microscopy. A system has been developed which has the capability to image domain structure while an in-plane magnetic field is applied *in situ*. All films exhibited a stripe domain structure in zero applied field which was indicative of a perpendicular component of domain magnetization which alternates in sign. All films showed a similar sequence of magnetization processes: on reducing the applied field from saturation a fine stripe domain structure nucleated and then coarsened as the field was decreased to zero. Local switching of domain contrast was observed along the steepest part of the hysteresis loop as the perpendicular component reversed. As the reverse field was increased toward saturation, the stripe domains disintegrated into smaller regions. This observation is consistent with an interpretation that the domain magnetization rotated locally into the sample plane. The saturation field and the film stress exhibited similar trends with nitrogen partial pressure. The results suggest that the perpendicular anisotropy that caused the formation of the stripe domain structure could be induced by the film stress via magnetoelastic coupling. [DOI: 10.1063/1.1344579]

I. INTRODUCTION

Thin films of soft magnetic materials, such as Sendust (FeSiAl), have applications as soft magnetic shield layers in magnetoresistive recording heads. In order to improve and optimize the soft magnetic properties of these types of films it is necessary to obtain a better understanding of how the microstructure and stress affect the magnetization processes and hence the magnetic properties. In this article we report on a study of the magnetization reversal of reactive rf-diode sputtered FeSiAl(N) films, using a magnetic force microscopy (MFM) with *in situ* applied field capabilities. By directly observing the domain structure under various applied fields for films whose stress and microstructure is well characterized, we hope to obtain a better understanding of the relationship between magnetic properties, stress, and microstructure.

As-deposited FeSiAl films sputtered with Ar usually have high coercivity, low permeability and large out-of-plane magnetic anisotropy.[1] Post-deposition annealing at elevated temperature around 400 to 600 °C is required to restore the ordered DO_3-type structure that gives soft magnetic properties.[1–3] Another way to achieve soft magnetic properties is to deposit Sendust films by N_2 reactive sputtering. It has been shown that addition of nitrogen to the sputtering gas can affect the grain size and stress of Fe alloy films and hence the magnetic properties.[4] Dodd *et al.* reported that soft Sendust films up to 500 nm thick can be deposited by N_2 reactive rf magnetron sputtering, and that a substantial decrease in coercivity was found when the ratio of nitrogen flow rate to N_2+Ar flow rate was 0.2%.[5] Nevertheless they observed an increase in coercivity and transition to columnar growth with increasing film thickness. Nanograined FeSiAl(N) films of several microns thick have been successfully prepared by dc magnetron sputtering[6] and rf-diode sputtering[7] using Ar+N_2 sputtering gas. In the latter study the deposited films exhibited a stripe domain structure, indicating the presence of a perpendicular anisotropy that could adversely affect the soft magnetic properties. The magneti-

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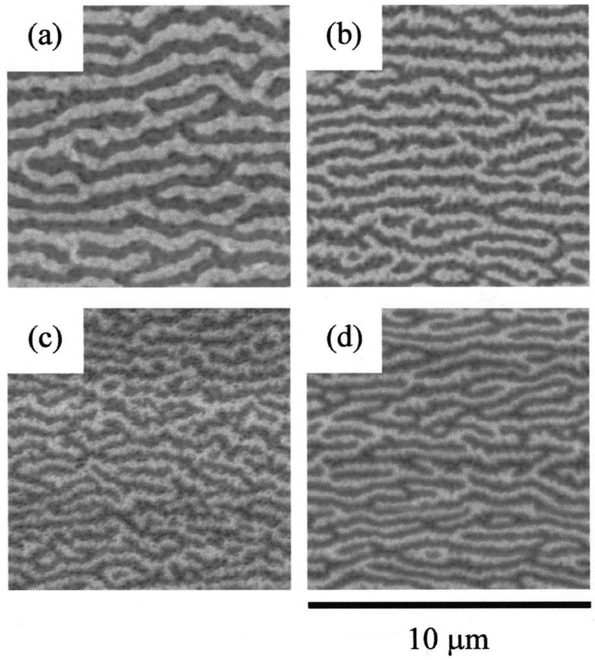

FIG. 1. MFM images obtained at the remanent state of the FeSiAl(N) films deposited with (a) 0%, (b) 3%, (c) 5%, and (d) 10% partial pressure of nitrogen in the sputtering gas.

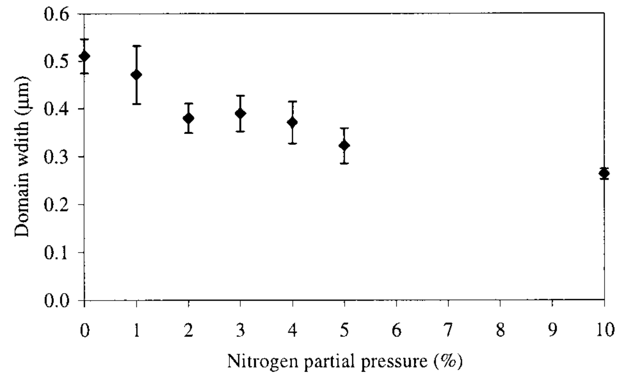

FIG. 2. Plot of the stripe domain width as a function of the partial pressure of nitrogen in the sputtering gas.

zation reversal in these FeSiAl(N) films is the subject of the present study.

II. EXPERIMENTAL DETAILS

A series of FeSiAl(N) films of thickness 1.7 μm were deposited by rf-diode sputtering onto Si(100) substrates with 300 nm of thermal SiO_2 on the surface. Sputtering gas was an $Ar+N_2$ mixture with different partial pressures (pp) of nitrogen ranging from 0% to 10%. Hysteresis loops were measured by vibrating sample magnetometry (VSM). Film microstructure was characterized by transmission electron microscopy (TEM). Film stress was determined by measuring the curvature of the film-substrate system using an atomic force microscope. Domain structure was studied using MFM with magnetic tips that were magnetized perpendicular to the sample plane. An electromagnet capable of producing an in-plane field up to 56 kA/m (700 Oe) was built and mounted on the sample stage. During the experiment samples were first demagnetized by applying an ac field with decaying amplitude along one direction. MFM images were then taken under various fields applied *in situ*. Reproducible image contrast was obtained from the same sample in the remanent state before and after the experiment, indicating that the tip was not remagnetized by the applied field.

III. RESULTS AND DISCUSSION

In zero applied field all films showed a stripe domain structure. Figure 1 shows the MFM images of some of the samples in the remanent state. The observed stripe domain pattern and the shape of the hysteresis loops (as shown in Figs. 3–5 for the 0%, 3%, and 5% pp N films, respectively) indicate that the adjacent stripes have aligned in-plane magnetization component while the perpendicular magnetization components alternate in sign. This domain configuration was first proposed by Saito *et al.* who observed stripe domains in Ni–Fe films using the Bitter method.[8,9] The alternating perpendicular magnetization component gave rise to the contrast of the MFM images since MFM measures the force gradient acting on the tip due to the perpendicular component of the surface field. In each sample the bright stripes and dark stripes were found to have similar widths, and reproducible images were obtained by rescanning the same area. These observations indicate that the domain structure of the films was not influenced substantially by the stray field from the tip. The width of the stripe domains were determined as half of the spatial period of the strongest component in the two-dimensional Fourier transform of the MFM images. As shown in Fig. 2, the domain width tends to decrease with increasing partial pressure of nitrogen in the sputtering gas during the film deposition.

Figure 3 shows the MFM images obtained from the same area of the 0% pp N sample at various stages of the hysteresis cycle. After the sample had been magnetized to saturation, on reducing the applied field a fine and irregular stripe domain structure nucleated [Fig. 3(b)]. The stripe domains coarsened and became more regular as the applied field was reduced to zero [Fig. 3(c)]. Along the steepest part of the hysteresis loop local switching of image contrast occurred, leading to connection and disconnection of the stripe domains [Figs. 3(c)–3(d)]. This suggests that the perpendicular magnetization component of parts of the stripe domains reversed. During this stage irreversible changes of the in-plane magnetization component also took place as indicated in the measured hysteresis loop. It was noticed that in this stage the domain width remained relatively constant and independent switching of image contrast of parts of a stripe domain were observed. These observations seem to suggest that the irreversible changes of the in-plane component occurred mainly by local switching of domain magnetization or by local motion of short sections of domain wall. This may be accompanied by switching the perpendicular component that was manifested as switching the image contrast. This magnetization reversal process is different from that brought about by simultaneous motion of long domain wall sections. In the latter case, domains of the preferred magnetization

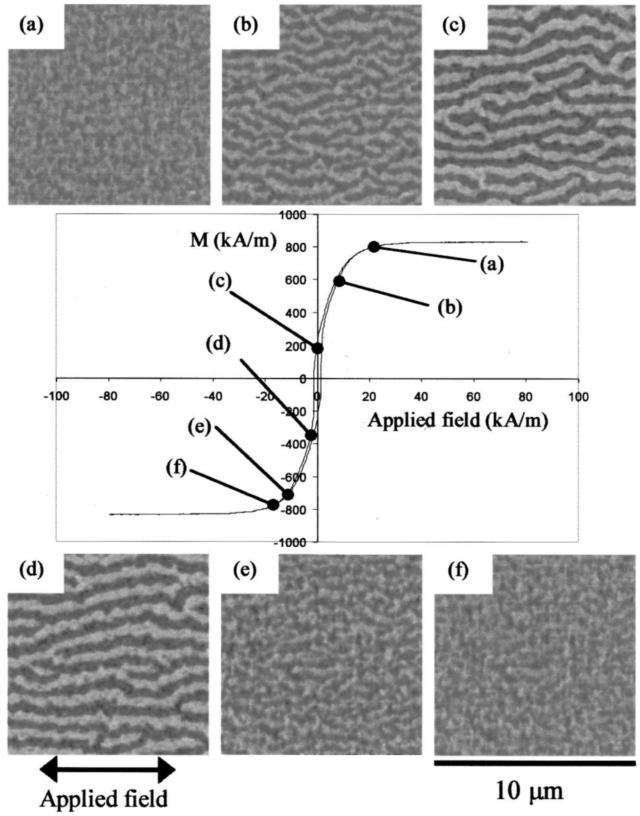

FIG. 3. MFM images obtained from the 0% pp N film in applied fields of (a) 19.9 kA/m, (b) 8.0 kA/m, (c) 0 kA/m, (d) −5.5 kA/m, (e) −15.9 kA/m, and (f) −20.0 kA/m.

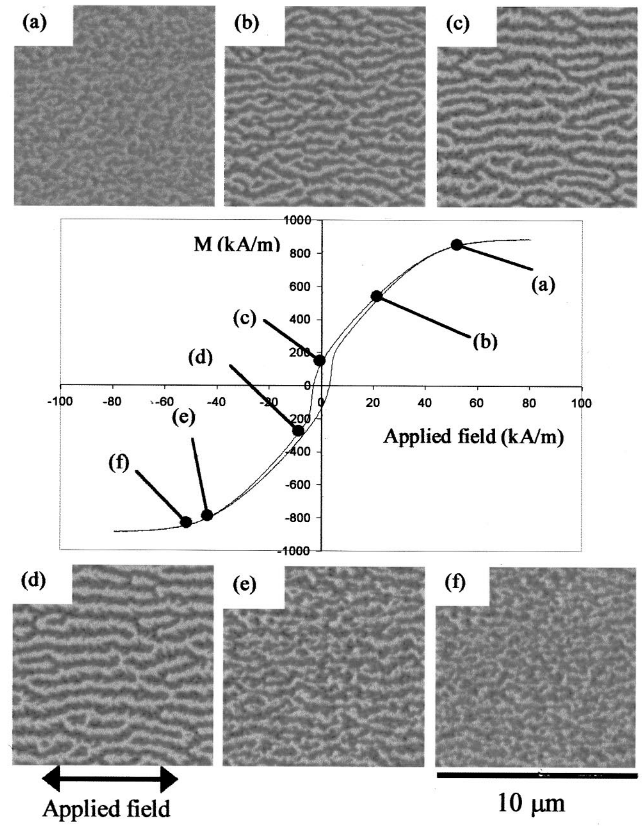

FIG. 4. MFM images obtained from the 3% pp N film in applied fields of (a) 51.8 kA/m, (b) 19.9 kA/m, (c) 0 kA/m, (d) −8.0 kA/m, (e) −43.8 kA/m, and (f) −51.8 kA/m.

direction would be observed to grow at the expense of neighboring domains as domain walls moved. Growing areas of the uniform MFM image contrast would be observed. This is contrary to the present observation that the stripe domain persisted and the domain width remained unchanged. The persistence of the stripe domain pattern could be due to the fact that the magnetostatic energy associated with it is lower than that of a uniformly magnetized domain that has a uniform perpendicular component.

As the reverse field was increased beyond the coercive field the stripe domains disintegrated into short and irregular segments [$<0.5\ \mu$m, Fig. 3(e)]. An interpretation of the observed change is that the process consists of local switching of domain magnetization of a few grains into the film plane (the grain size was measured to be about 0.1 μm by TEM). This process is hysteretic as indicated in the high-field regime (from about 1.2 to 16 kA/m) of the magnetization curve. The domain width was found to decrease when the applied field was increased beyond the coercive point as a result of the disintegration of the stripe domains. Similar variations in stripe domain width with applied field in the high field regime have been observed in previous studies on other Fe-based thin films.[10,11] Further increase in the applied field caused the image contrast to decrease as the in-plane magnetization along the field direction increased toward saturation [Fig. 3(f)].

Similar sequences of changes in MFM images were observed in the nitrided films. Nevertheless several differences in magnetization reversal between the 0% pp N film and the 1%–4% pp N films were noticed. For comparison the MFM images taken from the 3% pp N film are shown in Fig. 4. In the 3% pp N film a stripe domain structure was nucleated at a higher field than in the 0% pp N film [compare Fig. 4(a) with 3(a)]. A larger reverse field was needed for domain switching to take place in the 3% pp N film than in the 0% pp N film, showing that domain wall pinning is stronger in the former. Similar behavior was observed in the 1, 2, and 4% pp N films. These observations are consistent with the results of the VSM measurements which show that the 1%–4% pp N films had higher coercivities than the 0% pp N film.

The domain reversal of 5% and 10% pp N films exhibited subtle differences from that observed in the 0%–4% pp N films. As shown in Fig. 5, after stripe domains were nucleated [Fig. 5(b)] the domain pattern of the 5% pp N film exhibited smaller changes [Figs. 5(c) and 5(d)] than in the 0%–4% pp N films along the steepest part of the hysteresis loop. As the reverse field was increased beyond the coercive field the bright stripes became wider than the dark stripes. No disintegration of the stripe domains was observed when the sample magnetization approached saturation. Similar domain reversal was found in the 10% pp N film. This observation is in contrast to that made on the 0%–4% pp N films. A possible explanation is that in the high field regime the magnetization process taking place in the 5% and 10% pp N films involved mainly uniform rotation of domain magneti-

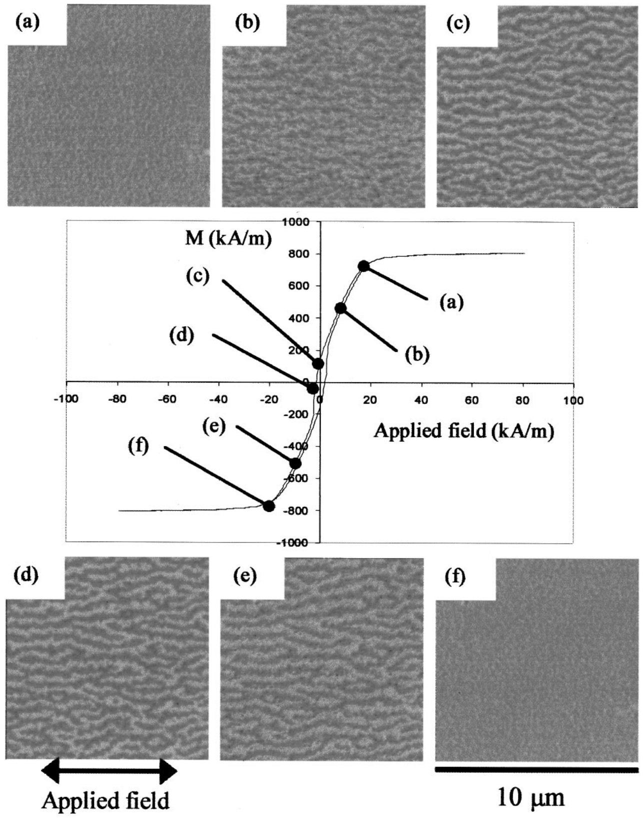

FIG. 5. MFM images obtained from the 5% pp N film in applied fields of (a) 19.9 kA/m, (b) 9.6 kA/m, (c) 0 kA/m, (d) −2.4 kA/m, (e) −10.0 kA/m, and (f) −19.9 kA/m.

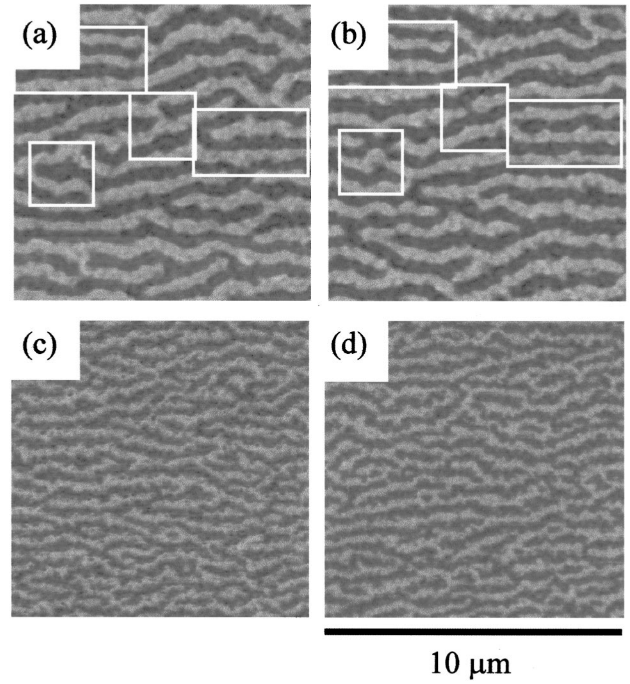

FIG. 6. MFM images obtained from the 0%pp N film at the oppositely magnetized remanent states are shown in (a) and (b). Notice the complementary contrast of the highlighted regions. The MFM images in (c) and (d) were obtained from the 5%pp N film at the oppositely magnetized remanent states.

zation towards the sample plane, while in the 0%–4% pp N films local switching of domain magnetization occurred instead.

Evidence of strong domain wall pinning was actually observed in the 0%–4% pp N films. An example is given in Figs. 6(a) and 6(b) which show the MFM images obtained from the 0% pp N sample at oppositely magnetized remanent states. Regions with complementary image contrast were observed. This indicates strong domain wall pinning, probably at the grain boundaries, as the domain width was of the same order of magnitude as the grain size. This suggestion is supported by the fact that strong domain wall pinning was not observed in the 5%–10% pp N samples. As shown in Figs. 6(c) and 6(d), the stripe domain structures found in the 5% pp N sample at the oppositely magnetized remanent states show much less repetition than in the 0% pp N sample. The observed difference in domain pinning between the two groups of films (namely the 0%–4% films, and the 5%–10% pp N films) could be related to the change in the film structure as pp N was increased from 4% to 5%. It was found in the TEM study that the 0%–4% pp N films have large columnar bcc grains (~0.1 μm), while the 5% and 10% pp N films consist of a mixture of randomly oriented equiaxed bcc nanograins (10 nm diameter or less) in an amorphous matrix (Fig. 7). Since the grains in the 5% and 10% pp N films are much smaller than the domain width, the effect of ripple and the strength of domain wall pinning are weaker than in the 0%–4% pp N films.

The stripe domain structure observed in all samples indicated a perpendicular anisotropy component that could be caused by the film stress via magnetoelastic coupling. If this is the case the strength of the applied field required to magnetize the sample to saturation against the perpendicular anisotropy (i.e., the saturation field) should depend on the film stress level. In this study the saturation field H_{sat} was determined by measuring the applied field at which the MFM image contrast reduced to 15% of its starting value at zero applied field. It was found that H_{sat} first increased with pp N, attained a maximum at 3% pp N and then decreased significantly for pp N$>$4% (Fig. 8). The film stress σ was found to be compressive for all samples, and the stress magnitude exhibited a trend with nitrogen partial pressure similar to that of H_{sat} as shown in Fig. 8. The close relationship between H_{sat} and σ tends to confirm that the perpendicular anisotropy is induced by the film stress caused by the presence of nitrogen.

For soft magnetic shield layer applications nanograined films, such as those produced near 5% pp N in the present study, should offer several significant advantages. The 5% pp N film has a saturation magnetization value similar to those of the 0%–4% pp N films. There is less domain pinning in the 5% pp N film since the grains are much smaller than the domain sizes. The effect of ripple, which is related to the variations in local anisotropy directions, and depends on the relative sizes of grain diameter and spin coupling length,[12] should also be much less than in the 0%–4% pp N films. Because of the random texture there is no magnetocrystalline contribution to perpendicular anisotropy in the 5% pp N film. The film has less stress and hence a lower

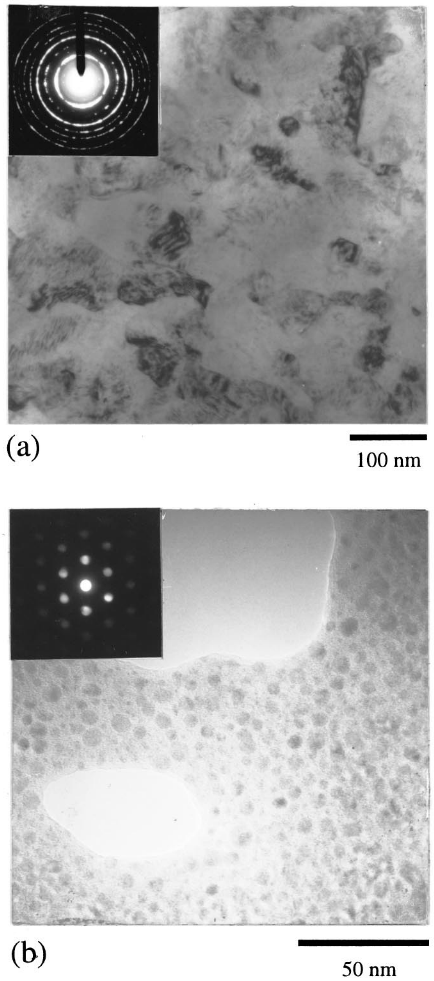

FIG. 7. Bright field TEM images obtained from (a) the 0%pp N film and (b) the 5%pp N film. The inset of (a) is the selected area diffraction pattern of the image. The inset of (b) is a convergent beam electron diffraction pattern showing the bcc structure of the nanograins.

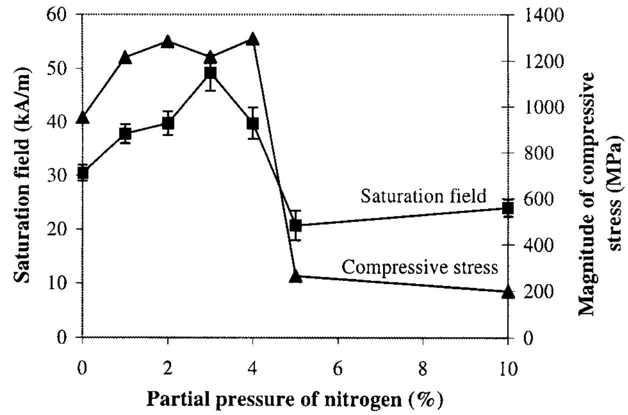

FIG. 8. Saturation field H_{sat} and the magnitude of the compressive film stress plotted as functions of the partial pressure of nitrogen.

saturation field (as shown in Fig. 8). However it still shows enough stress-induced perpendicular anisotropy to produce stripe domains.

Perpendicular anisotropy should be minimized as it adversely affects in-plane magnetic properties. The stripe domain structure should be avoided as it can make magnetization reversal more difficult. The film composition could be altered to give a negative magnetostriction so that an in-plane anisotropy would be induced by the compressive film stress. Alternatively the process could be optimized to decrease compressive stress or even make a small tensile stress.

IV. CONCLUSIONS

Magnetization reversal of a series of FeSiAl(N) films deposited using different partial pressures of nitrogen in the sputtering gas has been studied using a MFM with *in situ* applied field capabilities. Similar sequences of magnetization processes were observed in the films. All films of the series exhibit stripe domains. Substantial domain switching was found along the steepest part of the hysteresis loop. Strong domain wall pinning was observed in the 0%–4% pp N films, which have grain sizes of about 0.1 μm. This was not observed in the 5% and 10% pp N films which consisted of nanograins (10 nm diameter or less) in an amorphous matrix. The saturation field was found to be closely related to the stress level indicating that the perpendicular anisotropy is caused by the film stress via magnetoelastic coupling.

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